

**YTTRIUM OXIDE BASED SURFACE COATING
FOR SEMICONDUCTOR IC PROCESSING
VACUUM CHAMBERS**

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BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to equipment utilized in the manufacture of semiconductor integrated circuits (IC) and, in particular, to utilization of a yttrium oxide (Y_2O_3) coating on the anodized aluminum alloy components utilized in a semiconductor integrated circuit vacuum process chamber to improve corrosion resistance and erosion.

2. Discussion of the Related Art

Corrosion/erosion resistance is a critical property for parts used in IC fabrication vacuum chambers, where both corrosive chemistries and high-energy plasma bombardment reduce component lifetime and create contamination problems.

Anodized aluminum alloy is a primary material used in making components utilized in IC processing chambers. However, the high concentration of impurities contained in

conventional aluminum alloy causes formation of precipitates in the alloy which, in turn, cause internal cracks in the anodization layer. The integrity of the anodization layer is, thus, deteriorated and its corrosion resistance is severely damaged. The impurity concentration (wt.%) of "conventional" aluminum alloy, such as 6061, is as follows: Mg=0.8-1.20; Cu=0.15-0.40; Zn=max. 0.25; Mn= max. 0.15; Fe=max. 0.70; Si=0.40-0.80; Others=max. 0.15

More recently, high purity aluminum alloy material has been developed, resulting in minimal internal cracking in the anodization layer. Current data shows that chamber parts made from high purity aluminum alloy materials perform much better than those made from conventional alloy. "High purity" aluminum alloy means aluminum alloy with all impurities other than Mg being less than about 0.1 wt.% each, particularly Si, Fe and Cu.

Although high purity anodized aluminum alloy has a much better corrosion/erosion rate than the traditional anodized aluminum alloy, the anodization layer will still be attacked by the aggressive chamber environment after prolonged usage. The resulting need to replace parts reduces tool up-time and increases the cost of ownership.

Therefore there is always a need to continuously improve the lifetime of the aluminum alloy chamber components.

We are aware that yttrium oxide coatings have been used on anodized aluminum in the automobile and aerospace industries.

SUMMARY OF THE INVENTION

We have found that applying a ceramic-based surface protective layer, a yttrium oxide (Y_2O_3) coating, on the anodized surface of aluminum alloy chamber components improves the resistance of the anodized surface to corrosion and erosion by a factor of 5X over the anodized surface alone, particularly in the fluorine/oxygen plasma environment typically used in fabricating ICs.

A better understanding of the features and advantages of the present invention will be obtained by reference to the following detailed description and accompanying drawings that set forth an embodiment in which the principles of the invention are utilized.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1A is a SEM photograph showing a cross-section of a yttrium oxide coating on anodized aluminum alloy.

Fig. 1B is a SEM photograph showing a cross-section of a yttrium oxide coating on anodized aluminum alloy at higher resolution than the Fig. 1A SEM photograph.

Figs. 2A and 2B are SEM photographs showing, respectively, as-coated and after surface finished anodized aluminum alloy coated with yttrium oxide in accordance with the present invention.

DETAILED DESCRIPTION

Prototype vacuum chamber components made from
5 high purity anodized aluminum alloy (e.g., chamber liner,
cathode liner and door) were coated with various candidate
materials for testing with respect to erosion rate. A final
chamber test with coated parts was then conducted to verify
the performance of the coating. The result of chamber tests
10 showed that plasma spray coated Y_2O_3 had the lowest etch
rate among all tested materials.

Anodization of the aluminum alloy can be in
accordance with the disclosure of commonly-assigned and
co-pending U.S. patent application Serial No. (not yet
15 available), filed February 8, 2002, titled "Halogen-
Resistant Anodized Aluminum for Use in Semiconductor
Processing Apparatus" (Docket No. AM-6846) which
application is hereby incorporated by reference in its
entirety to provide additional background information
20 regarding the present invention.

More specifically, high purity 99.95% yttria was
sprayed on coupons made from anodized high purity
aluminum alloy material utilizing commercially available
plasma spray coating techniques. Other techniques for
25 applying the coating, e.g., chemical vapor deposition
(CVD) and physical vapor deposition (PVD), are also
appropriate.

Although the invention may be practiced utilizing
conventional aluminum alloy, utilization of high purity
30 aluminum alloy enables application of a barrier for the
applied coating.

The typical coating thickness is 5-7 mils. Important physical properties of the Y_2O_3 coating are listed as follows:

5	Composition	> 99.9% semiconductor Grade Y_2O_3
	Porosity	1 – 2% max.
	Density	5.0 g/cc
	Hardness	> 500 HV 0.3 (by cross-section)
10	Ra (μ in)	120
	CTE	8 – 9 μ m/m/ $^{\circ}$ C
	Thermal Conductivity	8 – 12 W/m. $^{\circ}$ K
	Coating Adhesion	3700 psi (on bare Al substrate)
		7100 psi (on anodized Al substrate)
15	Dielectric Constant	6-8 (ASTM D-159)

Fig. 1A shows the cross-section of an as-coated sample.

Fig. 1B shows a similar SEM cross-section taken at higher resolution. The Fig. 1A/1B photographs clearly show that the coating layer was dense, with all pores being isolated from each other. The maximum porosity was determined to be less than 1–2% based on the apparent metallographic method. The Y_2O_3 coating to high purity aluminum alloy substrate interface is intact. No delamination or other interfacial defects, such as voids and cracks, are observed.

It should be understood that up to about 10% Al_2O_3 can be combined with the Y_2O_3 to provide a Y_2O_3 -based coating with improved hardness and breakdown voltage characteristics.

Electrical properties were tested per ASTM standards in order to satisfy minimally required electrical properties for plasma chamber components. The results are listed below:

Breakdown Voltage of Y_2O_3 Coating (V)

	total voltage	voltage per mil
anodization (1 mil)	1,533	1,533
Y_2O_3 on bare Al (6 mil)	5,232	872
Y_2O_3 on anodized Al	5,894	843

Volume Resistivity (ohm-cm)

	25°C	60°C	150°C
anodization (1 mil)	7.4E15	4.1E15	4.9E15
Y_2O_3 on bare Al (6 mil)	1.5 E15	6.0E14	7.8E13
Y_2O_3 on anodized Al	9.8E14	4.1E14	3.4E13

The total breakdown voltage was found to be > 5 kv for a 6 mil coating layer, which was well above the 1kv criteria for a conventional anodization layer. The volume resistivity was also high enough to meet typical process requirements.

One major concern for the spray coated part is surface loose powders which were applied to the substrate during the very last spraying cycle. If these particles are not completely removed by a final finishing/cleaning process, these powders will have a high chance of coming off from the part's surface due to dynamic chamber conditions (erosion, corrosion, and thermal cycle). Once released to the chamber interior, they will cause a severe particle contamination problem.

To assure that no such loose particles are left on the yttrium oxide coating surface, the following methods are used.

First, a light mechanical finish is performed as part of the coating processes by manually holding a grinding tool over the as-coated surface, using silicon carbide (SiC) as the grinding medium. Fig. 2A and 2B are scanning electron micrographs that show as-coated and after surface furnished surface, respectively, clearly demonstrating that, after coating, the surface is very rough, with many powders

and nodules loosely attached to the surface. However, after finish, the coating surface is dense and free from loose particles.

After coating and surface finish, the coated parts are subjected to a standard cleaning procedure for chamber components that includes a CO₂ snow gun clean followed by a deionized (DI) water ultrasonic rinse performed at room temperature for about 15 minutes. All parts are then verified as particle-free by a Dryden QIII tool.

Cleanliness of the coating layer is another subject which requires close monitoring. Contamination can be introduced to the coating layer from various sources: raw powders, spray gun, process gases, and uncontrolled environment.

In the cleanliness study, the well-known Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) technique was used to determine the impurity levels in the coating layer. Table 1 below shows the impurity data of coating coupons. Two types of samples were used: after coating/finish from the coating vendor and after final clean from the cleaning vendor. Both samples were analyzed at surface and sub-surface for comparison. Each scan contained 60 pulses with 20 µm dia. spot size each pulse. The penetration depth was 1-2 µm. Sub-surface was achieved by pre-ablated top 1-2 µm layer away before testing.

There was some impurity difference between surface and sub-surface; in general, the sub-surface was found to be cleaner than the surface. Final clean reduced the coating impurity levels, indicating that the final clean process was effective to remove contaminants from the surface.

	after final clean		after coating and polish	
	surface	sub-surface	surface	sub-surf
Li	< 0.1	< 0.1	< 0.1	< 0.1
Be	< 0.1	< 0.1	< 0.1	< 0.1
Na	6	< 1	< 1	< 1
Mg	7.9	10.0	6.7	3.2
Al	123	70	415	213
Ca	< 1	< 1	< 1	< 1
Ti	12.4	3.6	60.0	8.0
Cr	7	7	1	< 1
Mn	< 1	< 1	1	11
Fe	1	< 1	< 1	60
Ni	1.6	1.0	0.3	0.1
Zn	75.1	63.4	88.0	19.9

Table 1: Y₂O₃ coating impurity analysis (ppm)

It should be understood that various alternatives to the embodiments of the invention described herein may be employed in practicing the invention. Further example, as discussed above, it should be understood that in addition to high purity 99.95% yttria, any yttria based coating will provide erosion/resistance enhancements over anodization alone. Thus, it is intended that the following claims define the scope of the invention and that methods and structures within the scope of these claims and their equivalents be covered thereby.